1 Introduction

W-ZrC cermet is an important material for high temperature structural applications such as in aerospace, automobile and electronic industry because of its superior high temperature strength and high elastic modulus. Therefore, extensive research has been conducted towards understanding the mechanical properties and thermophysical properties in different environments. However, few studies of the coarsening phenomena of the W-ZrC cermet were reported although it is important to performance such as sustainability and durability. In this study, powder mixtures of W-x vol.% ZrC (x=10 to 30) were prepared using commercial ZrC and as-reduced ZrC at 1400°C for 2hrs and followed by spark plasma sintering to reduce the sintering time. [ref. 1,2,3]

2. Experimental Procedures

As a starting material, 2 types of powder mixtures were prepared by ball-milling with WC balls and polythene jar in ethanol. The first type of powder mixture, namely W-ZrC (SNU), was mixed with a commercially available pure tungsten powder (2.3 μm, TaeguTec, Seoul, Korea) used as a matrix material and ZrC powder carbothermally reduced in a vacuum furnace at 1400°C for 2hrs and followed by spark plasma sintering to reduce the sintering time. [ref. 1,2,3]

The apparent density of the sintered specimens was measured using the Archimedes method in water. Microstructure of the samples was examined using a scanning electron microscope (Normal SEM 6360, JEOL, Japan) with back scattered electron mode after polishing the surfaces of specimens by using a diamond suspension of 6 μm and 1 μm. The elastic modulus (E) was determined by an ultrasonic pulse-echo testing (Tektronix TDS 220, Panametrics, Model 5800, Korea). Vickers Hardness (Mitutoyo, Japan) was measured at 20 kg load for 15 s, while fracture toughness was estimated from the crack length measurements based on Anstis’s formula after indenting at 20 kg load for 15 s. [ref. 4]

To investigate a coarsening phenomenon of the composites, the 2 specimens among sintered W-ZrC
(SNU) samples and sintered W-ZrC (Com) samples whose the apparent density was the maximum at the same sintering temperature were chosen respectively and cut into 4 equal parts. The divided parts were heat-treated again in vacuum furnace at 1550°C for 0.5 h, 1 h, 2 hrs and 3 hrs, respectively. Microstructures and mechanical properties of the samples was re-examined ditto.

3. Results

XRD patterns of the developed W-ZrC specimens sintered at 1850°C for 0min are shown in Fig. 1. For comparison, the pattern of the sintered sample with various contents of ZrC was included from 0 vol.% to 30vol.% ZrC which can be attributed to the presence of W2C second phase. With small portion of ZrC contents, W2C phase almost completely disappeared and nearly desirable phases such as W and ZrC were obtained. It is the fact that the carbon diffusion from a carbon mold is not serious rather, W2C phase was produced because of ZrC particles because the peak shifts from lower angles to higher angles were found on ZrC peaks. It means the lattice parameter of ZrC decreased by substituting Zr4+ with W4+. [ref. 5,6,7]

The sintering temperature was ranged from 1700 to 1900°C for 0~10 min. Desirable density values were obtained above 1850°C for 0min. W-x vol.%ZrC (x=10 and 30) specimens were post-annealed at 1550°C for 3hrs in a vacuum furnace after SPS process. In the microstructure, the size of coalesced ZrC particles was increased and the shape of the particles became more faceted than that of previous ones by the post heat treat. Density and modulus increased after post heat treatment, while hardness decreased. Mechanical and physical properties (density, modulus and hardness) were related to the microstructure and processing conditions. [ref. 8,9,10]
Fig. 2 The evolution of apparent density of all the samples

There was an increase in hardness. It was attributed to that the sinterability was elevated by the formation of (Zr, W)C phase and that the W₂C phase harder than monolithic W was produced unexpectedly in sintering process. It is also interesting that deviation of hardness on W-xvol.%ZrC (Com, X=10 and 30) was larger than 3~8 times compared with that of hardness on W-xvol.%ZrC (SNU, X=10 and 30). These results indicated the microstructures were not homogeneous, thus hardness was also different in some microstructures. After annealing, it was found that there was a little decrease in hardness and this result followed Hall-Petch relationship. It was also thought that the tendency of deviation of hardness was similar to the above tendency of results although the samples were post-heat treated. As a result, the microstructures were affected by ZrC (Com) powder supposed to be more unstable than ZrC (SNU), thus finally hardness was different locally.

Fig. 3 The change of hardness with annealing time increase

The elastic modulus of the developed W-ZrC composites varied in the wide range of 350~400 GPa (Table 2). This result indicated the addition of ZrC could be effective on the strength of the developed W-ZrC composites because the elastic modulus is related more or less with the strength. It was interesting that the increase of the elastic modulus was not proportional to the content of ZrC. The maximum value of elastic modulus was found on the W-15vol.%ZrC though the value of W-15vol.%ZrC was not described in Table 2 and then an decreasing tendency was shown as the content of ZrC increased. It was also known that the tendency of the elastic modulus followed accurately that of the apparent density. That’s why the elastic modulus was highly elevated after annealing. As above mentioned, the maximum density was found on the W-15vol.%ZrC composite.

Fig. 4 The evolution of modulus with time

The fracture toughness was measured using indentation fracture toughness methods but crack propagation was not occurred because of ductility of W matrix except for the developed W-30vol.%ZrC (SNU) composites. It was supposed that brittleness of the developed W-30vol.%ZrC (SNU) composites increased as an amount of ZrC increased and the microstructures of W-30vol.%ZrC (SNU) composites were more homogeneous.

Fig. 5 The change of toughness of the W-30vol.%ZrC(SNU) sample that cracks were found only with annealing time increasing
Although all the sintered samples were annealed again, crack propagation was discovered on the only W-30vol.%ZrC (SNU) composite. The fracture toughness was improved with increasing grain size. It was reported that W-30vol.%ZrC composites sintered at 1900°C−2000°C for 1h−2hrs under vacuum condition had a elastic modulus of ~380 GPa, a hardness of 5−6 GPa and a fracture toughness of ~9 MPa-m^{1/2}. [ref. 2,6,8,9]

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References


